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# Response to Comments on the application of the Scherrer equation in "Copper aluminum mixed oxide (CuAl MO) catalyst: A green approach for the one-pot synthesis of imines under solvent-free conditions", by Suib et al. [Appl. Catal. B: Environ, 188 [2016] 227–234, doi:10.1016/j.apcatb.2016.02.007]

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ABSTRACT

This paper concerns data analysis using X-ray line broadening and electron microscopy methods. The data reported here show that electron microscopy data are preferable in terms of determining a size distribution and an average size for these materials.



We thank Morato and Rives [1] for pointing out problems in the data analyses of X-ray line broadening (XRLB) experiments in our publication [2]. New data and calculations on these materials are included here that are more reasonable. We will note specific problems in the data analyses of these systems. First of all, there are about 60 entries for copper(II) oxide in the JCPDS X-ray data base. Of these, approximately 20 entries are for tenorite, the material in question. Clearly this is a complicated material. Our analyses suggest that JCPDS card number is 01-073-6372, the best fit to experimental data. Secondly, Morato and Rives [1] point out that several of the peaks are convoluted and impossible to separate. Such situations make XRLB calculations quite complicated. JCPDS card 01-073-6372 suggests that the diffraction intensity at 35.56° 2 theta is only due to the (002) reflection in contrast with suggestions of Morato and Rives [1], but that the intensity at 38.76° two theta is due to two reflections (111) and (200) as they suggest. Accordingly, Fig. 1 in the original paper should be replaced by the following Fig. 1.

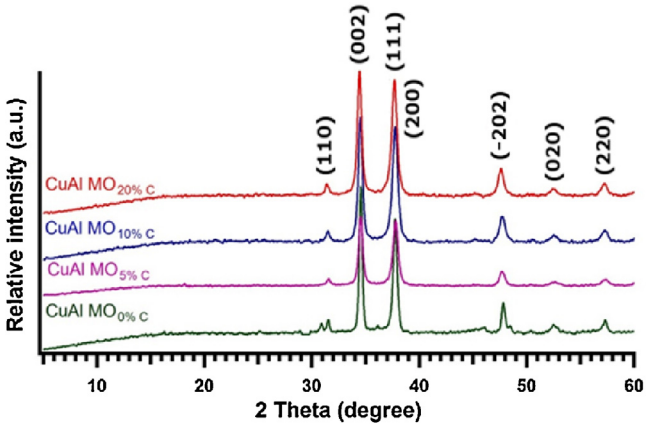


**Fig. 1.** X-ray diffraction patterns of CuAl MOs synthesized with different amounts of activated carbon.

In terms of XRLB analyses of the above data, we originally used all of the peaks for the analysis (not just the most intense peak as assumed by Morato and Rives [1]). The Rigaku program PDXL was used for these analyses and we were able to reproduce the data originally reported. Clearly, however, there is a problem using that program in obtaining meaningful data. In fact, we did XRLB calculations with the program using all sorts of combinations of the most intense peaks, all peaks, individual peaks, and using both the Halder Wagner method as well as the Hall method and the results are not meaningful as regards particle size distribution data from transmission electron microscopy (TEM) results (*vide infra*). Hand calculations using methods similar to Morato and Rives[1] also are not in agreement with TEM data. Clearly when structures are not well defined, peaks overlap, and there are small particles, XRLB is not the method of choice in determination of average particle sizes. In fact, we have published a paper here warning about discrepancies between XRLB and TEM data for nano-size particles [3].

Nevertheless, even though particle size has little to do with the original article, a proper particle size distribution for these systems is warranted. TEM data and particle size distributions for the samples reported in the original paper are shown in Fig. 2. The average particle size for CuO itself (Fig. 2a) is ~200 nm. Similar experiments were done for CuAl MO 5%C (Fig. 2b and e) and the CuAl MO 10%C (Fig. 2c and f) and CuAl MO 20%C (Fig. 2d and g) samples of the original paper yielded average particle of 59 nm, 29 nm, and 30 nm, respectively. These average particle sizes should replace those of Table 1 Column 2 in the original paper with the caveat that they were determined by TEM. Fortunately, as pointed out by Morato and Rives [1], this mistake in data analysis has no effect on the rest of the paper.

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0926-3373/

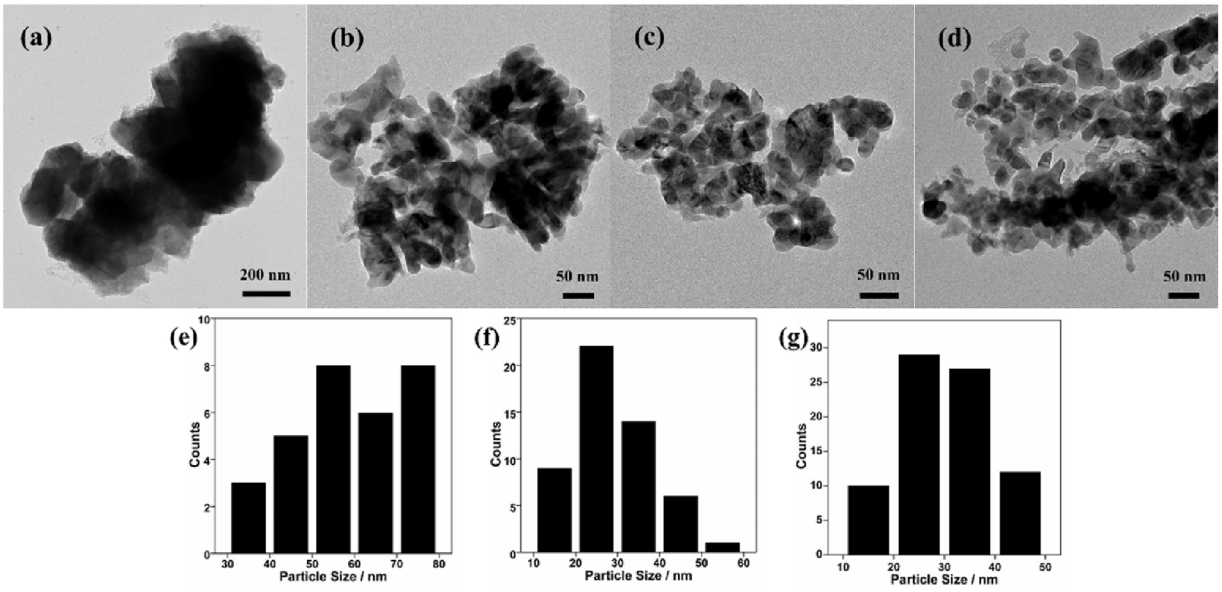


**Fig. 2.** TEM Images and Particle Size Distributions for CuAl MO (a), CuAl MO 5%C (b and e), CuAl MO 10%C (c and f), and CuAl MO 20%C (d and g).

## References

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